ARCHIVES OF ACOUSTICS
4, 2, 109-116 (1979)

ACOUSTICAL INVESTIGATIONS OF THE EFFECT OF ADDITIVES ON THE ELASTIC PROPERTIES OF RAW KAOLIN*

EWA RYLL-NARDZEWSKA, JAN RYLL-NARDZEWSKI, ANDRZEJ KULIK

Department of Physical Acoustics, Institute of Fundamental Technological Research
00-049 Warszawa, ul. Świętokrzyska 21

and HENRYK PIECZAROWSKI

Institute of Glass and Ceramics, Pruszków Branch
05-800 Pruszków, ul. 17 Stycznia 77

To improve the mechanical strength of ceramic bodies various modifying agents are added in the raw state. The effect of the modifiers on the elastic properties of kaolin samples in the raw state and after firing was studied by acoustical methods: a resonance one and a pulse one. The results of the Young's modulus measurements obtained by the acoustical methods agree with the results obtained by other methods. The resonance methods have been found to be applicable for the examination of raw ceramic materials.

1. Introduction

The mechanical strength and plasticity of raw kaolin materials are often insufficient to form a sample into a desired shape. A low mechanical strength in the dried semi-finished products is undesirable since they are liable to be damaged during the subsequent production stages. To reduce the amount of damage, various modifying additives are added to the casting slip in order to improve the plasticity and mechanical strength of the semi-finished ceramic products.

* Supported by the interdisciplinary contract No. MR-I.24.

The paper presents the results of preliminary investigations of the effect of modifiers on the elastic properties of raw and fired kaolin. The investigations were performed using non-destructive acoustical methods: a pulse one [1] and a resonance one [2]. The measurements were performed at the Institute of Fundamental Technological Research of the Polish Academy of Sciences. The samples were prepared at the Institute of Glass and Ceramics at Pruszków.

2. Sample preparation

The samples to be tested were prepared from KOC kaolin of the following physical and chemical properties:

(a) chemical composition [%]

firing losses	11.54	CaO	0.36
SiO_2	52.95	MgO	0.48
Al_2O_3	33.15	Na_2O	0.06
Fe_2O_3	0.58	K_2O	0.69
TiO_2	0.49		

(b) mineral composition [%]

clay minerals	83.28
quartz	12.04
feldspar	4.68

(c) particle-size analysis

dimension [μm]	[%]	dimension [μm]	[%]
> 60	1	7-5	94.0
60-30	99	5-3	77.1
30-20	98.7	3-2	67.2
20-10	94.0	> 2	56.7

The following modifiers were added to the KOC kaolin:

(a) Rokrysol WF-1 (coagulum manufactured by "Rokita", Brzeg),

(b) Glycocel (carboxymethylcellulose sodium),

(c) Ground bentonite (Milowice).

The casting slip was prepared by first pouring the modifier into water. After mixing, dry KOC kaolin was added, mixed for 2 hours with a stirrer and

left to stand for 24 hours. The suspension thus prepared was mixed, separated from the sludge using No. 1 Sieve (3600 holes/cm²) and filtered using a laboratory filter press operated at a pressure of 10^6 N/m². The discs obtained were de-aerated in a vacuum press to homogenize the composition and next formed in the shape of samples in the same press. Next the samples were dried at 110°C to the surface-dry state. After drying, some of the samples were fired in a silit chamber kiln at 1250°C.

3. Measurement method

The investigated samples had the form of rods several centimeters long and a square cross-section of about 1 ×1 cm. Such a form for the samples made it possible to perform the measurements on both a concrete tester and a resonance elastometer. In some cases the form of the samples deviated markedly from the cubicoid. However, in spite of that, meaningful preliminary results were obtained on the effect of some additives on the mechanical properties of kaolin.

Raw samples were examined using a BI-8R-M66 Ultrasonic Concrete Tester manufactured by "Radiotechnika" (Wrocław). The measurements were performed using a transmission method at frequencies of 250 kHz and 500 kHz generated by G-250 and G-500 heads. The fired samples were tested using a Unipan 541 Material Tester and 100 kHz 100 LBN1 ultrasonic heads. The samples were coupled with transducers using "Epidian 5" epoxyadhesive.

In making the measurements by an acoustical resonance method a modern version of a resonancee elastometer, produced by IFTR PAS (Warsaw), was used [3]. This instrument is provided with capacitance type, contactless electroacoustic transducers and, therefore, the ceramic samples to be examined were covered with a thin silver layer. The layer is several micrometers thick and does not affect the measurement results. The samples excited in a longitudinal mode. The principle of the measurement is illustrated in Fig. 1. A transmitting electrode

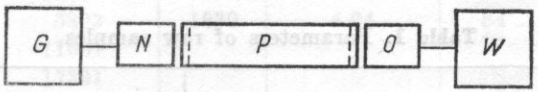

Fig. 1. Schematic diagram of the resonance elastometer
G – generator, *N* – transmitting transducer, *P* – sample, *O* – receiving transducer *W* – amplifier

N is biased with a D. C. voltage and fed with a signal from a variable frequency sine wave generator *G*. By varying the generator frequency *f* one can excite successive resonances in the sample (cf. Table 3). The oscillations of the sample *P* are detected by a receiving transducer *O*. Next the signal from the transducer is supplied to an amplifier *W*. The frequency of the resonance oscillation is measured with a digital frequencymeter built into the measuring system. The propagation velocity in the samples is determined from the relation

$$c = \lambda_n f_n, \tag{1}$$

where c is the velocity of the elastic wave, λ_n – the wavelength, and f_n – the frequency of the n-th resonance.

The wavelength in the case of longitudinal modes is

$$\lambda_n = \frac{2}{n} l, \tag{2}$$

where n is the order of the resonance, and l – the sample length.

In the case of slender samples where the length l is much greater than the transverse dimension and the latter is much smaller than the wavelength, the propagation velocity is expressed by the relation

$$C = \sqrt{\frac{E}{\varrho}}, \tag{3}$$

where E is the Young's modulus, and ϱ – the specific density of the material.

4. Results

The results of measurements and the parameters of samples are summarized in Tables 1-4 and in Figs. 2 and 3. The results obtained indicate that the additives denoted in the figures by R (rokrysol), G (glycocel) and B (bentonite) affect the propagation velocity, and thus the Young's modulus, to various degrees. Agreement between the results obtained by the different measurement methods (the pulse method and the resonance method) is good.

The measurements performed on the raw samples reveal a dispersion of the ultrasonic wave velocity brought about by the unfavourable ratio of the transverse dimension of the samples and the wavelength [4]. The dispersion of the ultrasonic wave velocity observed is also due to the large inhomogeneity of the structure.

Table 1. Parameters of raw samples

Sample No	Dimensions l [m]	$b \times h$ [m]	ϱ [kg·m^{-3}]	Materials
1	0.1475	0.011 × 0.11	1480	kaolin
2	0.1353	0.011 × 0.011	1610	kaolin +0.2% rokrysol
3	0.1298	0.011 × 0.011	1500	+0.5% rokrysol
4	0.1407	0.011 × 0.011	1430	+1.0% rokrysol
5	0.1404	0.011 × 0.011	1520	+0.2% glycocel
6	0.1525	0.011 × 0.011	1590	+0.5% glycocel
7	0.1521	0.011 × 0.011	1510	+1.0% glycocel
8	0.1187	0.011 × 0.011	1560	+0.2% bentonite
9	0.1488	0.011 × 0.011	1540	+0.5% bentonite
10	0.0947	0.011 × 0.011	1530	+1.0% bentonite

Table 2. Parameters of fired samples (firing temperature 1523 ± 20 K)

Sample No	Dimensions			Materials
	l [m]	$b \times h$ [m]	ϱ [kg·m^{-3}]	
1	0.0547	0.0096 × 0.0096	2270	kaolin
2	0.1260	0.0097 × 0.0101	1910	kaolin +0.2 % rokrysol
3	0.0708	0.0097 × 0.0101	1780	kaolin +0.5 % rokrysol
4	0.1283	0.0097 × 0.0098	1730	kaolin +1.0 % rokrysol
5	0.0588	0.0096 × 0.0099	1830	kaolin +0.2 % glycocel
6	0.1416	0.0099 × 0.0099	1830	kaolin +0.5 % glycocel
7	0.0826	0.0099 × 0.0099	1740	kaolin +1.0 % glycocel
8	0.1099	0.0099 × 0.0100	1840	kaolin +0.2 % bentonite
9	0.1388	0.0099 × 0.0100	1820	kaolin +0.5 % bentonite
10	0.0875	0.0099 × 0.0100	1910	kaolin +1.0 % bentonite

Table 3. The results of measurements on the raw samples

Sample No	Resonance method		Pulse method f = 250 kHz		
	Resonance frequency [Hz]	c^*_{av} [m/s]	E [N/m^2]·10^9	τ [μs]	c [m/s]
1	5018 10072 15106	1485	3.26	98	1505
2	6406 12728 19078	1725	4.80	78	1735
3	5885 11798 17636	1530	3.51	50	1550
4	5229 10452 15724	1470	3.10	94	1495
5	5803 11661 17391	1630	4.04	84	1670
6	5964 11904 17845	1815	5.24	82	1860
7	5967 11841 17755	1805	4.92	83	1830
8	6293 12437 18744	1485	3.44	76	1560
9	5434 10805 16230	1605	3.90	90	1655
10	8970 17935 26679	1695	4.39	56	1690

* c_{av} was determined as the mean value for the three modes

Table 4. The results of measurements on the fired samples (firing temperature 1523 ± 20 K). The results were obtained by the pulse method at a frequency of 100 kHz

Sample No	τ [μs]	c [m/s]	E [N/m²]·10⁹
1	12.5	4370	43.3
2	27.9	4515	38.9
3	16.4	4315	33.1
4	31.2	4110	29.2
5	13.4	4375	35.2
6	32.1	4410	35.6
7	19.6	4225	31.1
8	26.5	4145	31.6
9	33.7	4120	30.9
10	20.0	4310	35.5

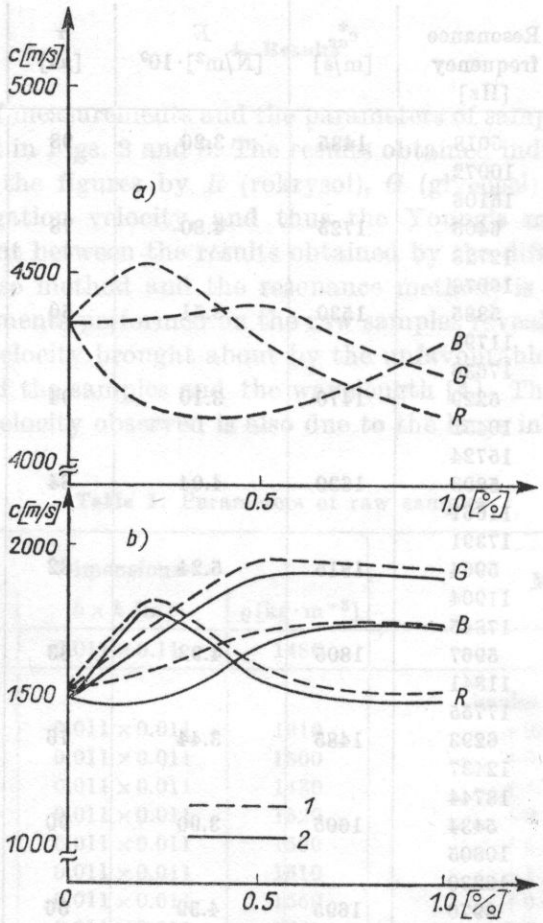

Fig. 2. The relation of the propagation velocity to the additive concentration in kaolin
a) fired samples, b) raw samples; R – rokrysol, G – glycocel, B – bentonite

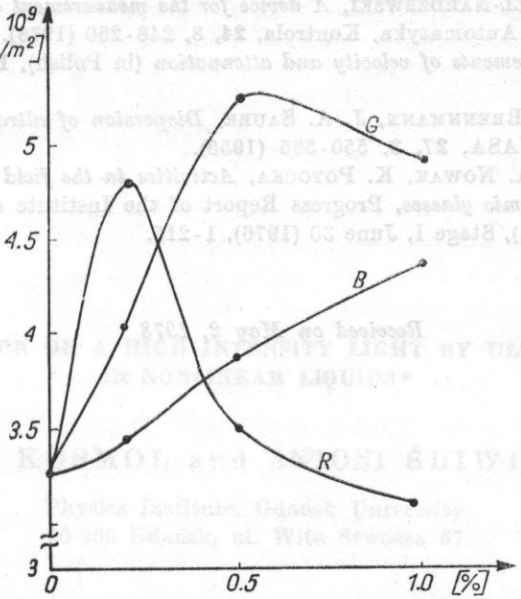

Fig. 3. The relation of value of Young's modulus E to the amount of modifier in the raw samples, determined by the acoustic resonance method
R – rokrysol, G – glycocel, B – bentonite

In the resonance method the measurements are performed in a range in which the transverse dimension of the sample is much smaller than the wavelength and, therefore, one can assume that the wave propagation in the sample is of the rod type [3].

4. Conclusions

The investigations performed indicate that acoustical methods and, in particular, the acoustic resonance method can be used for the determintation of the strength characteristics of ceramic clay materials in the raw state.

The results of the Young modulus mesaurements obtained by acoustical methods agree with the results of plasticity measurements performed in the Laboratory of Rheological Investigations of the Institute of Glass and Ceramics, Pruszków Branch [6].

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Received on May 2, 1978